

ADDITIVE COMPOSITION FOR TRANSMISSION OIL

The present invention is directed to an additive composition for a transmission oil. More particularly, the present invention is directed to an additive composition comprising an oil dispersion of hexagonal boron nitride and a viscosity index improver, in particular, an additive composition containing a viscosity index improver
5 selected from a polymethacrylate, a dispersant polymethacrylate or a dispersant olefin copolymer.

REFERENCES

10 The following references are cited in this application as superscript numbers:

¹ Peeler, U.S. Patent No. 3,313,727, *Alkali Metal Borate E.P. Lubricants*, issued April 11, 1967

² Adams, U.S. Patent No. 3,912,643, *Lubricant Containing Neutralized*
15 *Alkali Metal Borates*, issued October 14, 1975

³ Sims, U.S. Patent No. 3,819,521, *Lubricant Containing Dispersed Borate and a Polyol*, issued June 25, 1974

⁴ Adams, U.S. Patent No. 3,853,772, *Lubricant Containing Alkali Metal Borate Dispersed with a Mixture of Dispersants*, issued December 10, 1974

20 ⁵ Adams, U.S. Patent No. 3,997,454, *Lubricant Containing Potassium Borate*, issued December 14, 1976

⁶ Adams, U.S. Patent No. 4,089,790, *Synergistic Combinations of Hydrated Potassium Borate, Antiwear Agents, and Organic Sulfide Antioxidants*, issued May 16, 1978

25 ⁷ Adams, U.S. Patent No. 4,163,729, *Synergistic Combinations of Hydrated Potassium Borate, Antiwear Agents, and Organic Sulfide Antioxidants*, issued August 7, 1979

⁸ Frost, U.S. Patent No. 4,263,155, *Lubricant Composition Containing an Alkali Metal Borate and Stabilizing Oil-Soluble Acid*, issued April 21, 1981

⁹ Frost, U.S. Patent No. 4,401,580, *Lubricant Composition Containing an Alkali Metal Borate and an Ester-Polyol Compound*, issued August 30, 1983

5 ¹⁰ Frost, U.S. Patent No. 4,472,288, *Lubricant Composition Containing an Alkali Metal Borate and an Oil-Soluble Amine Salt of a Phosphorus Compound*, issued September 18, 1984

¹¹ Clark, U.S. Patent No. 4,534,873, *Automotive Friction Reducing Composition*, issued August 13, 1985

10 ¹² Brewster, U.S. Patent No. 3,489,619, *Heat Transfer and Quench Oil*, issued January 13, 1970.

¹³ Salentine, U.S. Patent No. 4,717,490, *Synergistic Combination of Alkali Metal Borates, Sulfur Compounds, Phosphites and Neutralized Phosphate*, issued January 5, 1988

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All of the above patents are herein incorporated by reference in their entirety to the same extent as if each individual patent was specifically and individually indicated to be incorporated by reference in its entirety.

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BACKGROUND OF THE INVENTION

High load conditions often occur in gear sets such as those used in automobile transmissions and differentials, pneumatic tools, gas compressors, centrifuges, high-pressure hydraulic systems, metal working and similar devices, as well as in many types of bearings. When employed in such environments, it is conventional to add an extreme-pressure (E.P.) agent to the lubricant composition and, in this regard, alkali metal borates are well known extreme-pressure agents for such compositions.^{1-11, 13} E.P. agents are added to lubricants to prevent destructive metal-to-metal contact in the lubrication of moving surfaces. While under normal conditions termed

25 “hydrodynamic”, a film of lubricant is maintained between the relatively moving surfaces governed by lubricant parameters, and principally viscosity. However, when load is increased, clearance between the surfaces is reduced, or when speeds of moving surfaces are such that the film of oil cannot be maintained, the condition of

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“boundary lubrication” is reached; governed largely by the parameters of the contacting surfaces. At still more severe conditions, significant destructive contact manifests itself in various forms such as wear and metal fatigue as measured by ridging and pitting. It is the role of E.P. additives to prevent this from happening. For the most part, E.P. agents have been oil soluble or easily dispersed as a stable dispersion in the oil, and largely have been organic compounds chemically reacted to contain sulfur, halogen (principally chlorine), phosphorous, carboxyl, or carboxylate salt groups which react with the metal surface under boundary lubrication conditions. Stable dispersions of hydrated alkali metal borates have also been found to be effective as E.P. agents.

Moreover, because hydrated alkali metal borates are insoluble in lubricant oil media, it is necessary to incorporate the borate as a dispersion in the oil and homogenous dispersions are particularly desirable. The degree of formation of a homogenous dispersion can be correlated to the turbidity of the oil after addition of the hydrated alkali metal borate with higher turbidity correlating to less homogenous dispersions. In order to facilitate formation of such a homogenous dispersion, it is conventional to include a dispersant in such compositions. Examples of dispersants include lipophilic surface-active agents such as alkenyl succinimides or other nitrogen containing dispersants as well as alkenyl succinates.^{1-4, 12} It is also conventional to employ the alkali metal borate at particle sizes of less than 1 micron in order to facilitate the formation of the homogenous dispersion.¹¹

In addition, anti-sticking agents are often employed in automotive gear boxes to provide smooth synchronization and good shift ability. Examples of such anti-sticking agents include phosphates, phosphites, phosphonates, thiophosphates, carbamates, molybdenum dithiocarbamates and dithiophosphates.

It is also known that boron nitride exhibits friction modifying properties in lubricants. For example, U.S. Patent No. 4,787,993, issued November 29, 1988 to Nagahiro, discloses a lubricant effective for the reduction of friction which comprises dispersing a finely powdered aromatic or polyamide resin into a fluid fat or oil, which may additionally contain molybdenum disulfide, organic molybdenum or boron nitride.

Furthermore, U.S. Patent No. 4,715,972, issued December 29, 1987 to Pacholke, discloses a solid lubricant additive for gear oils comprising solid lubricant particles combined with a stabilizing agent and a fluid carrier, wherein the solid lubricant particles are selected from the group consisting of molybdenum disulfide, graphite, cerium fluoride, zinc oxide, tungsten disulfide, mica, boron nitrate, boron nitride, borax, silver sulfate, cadmium iodide, lead iodide, barium fluoride, tin sulfide, fluorinated carbon, PTFE, intercalated graphite, zinc phosphide, zinc phosphate, and mixtures thereof. This patent further discloses that such lubricant additive provides the gear oil with improved demulsibility, stability and compatibility characteristics of the gear oil when contaminated with water.

Polymethacrylic acid esters or polymethacrylates are long chain esters commonly used in the lubricating oil industry as viscosity index improvers (VII). Their molecular masses lie predominantly between 20,000 and 500,000. The properties of the homo- or co-polymers of the various alkylmethacrylates differ with the chain length of the alcohol used to make the ester and the degree of polymerization. Olefin co-polymers (OCP) are manufactured from ethylene and propylene by means of Ziegler catalysts and are commonly used in the lubricating oil industry as VIIs. Dispersant Olefin Co-polymers (DOCP) are multifunctional VIIs; the viscosity improving effect is combined with dispersant properties by the inclusion of cyclic imides such as N-vinylimidazole and similar fragments in the polymers.

Accordingly, it is an object of the present invention to provide a lubricant additive composition having good anti-sticking properties when used in transmission oils.

SUMMARY OF THE INVENTION

The present invention provides a novel additive composition for a transmission oil comprising:

- a) an oil dispersion of hexagonal boron nitride and;
- b) a viscosity index improver selected from the group consisting of:
 - i) a polymethacrylate,
 - ii) a dispersant polymethacrylate, and

iii) a dispersant olefin copolymer;
wherein the weight ratio of the oil dispersion of hexagonal boron nitride to the viscosity index improver is in the range of from about 99:1 to about 1:99.

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Typically, the concentration of the oil dispersion of hexagonal boron nitride is from about 1 to about 99 wt %, preferably from about 5 to about 95 wt % and the concentration of the viscosity index improver is from about 1 to about 99 wt %, preferably from about 5 to about 95 wt %, based on the total weight of the additive composition.

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The additive composition of the present invention may optionally further contain an oil dispersion of hydrated alkali metal borate containing a hydrated alkali metal borate, a dispersant, optionally a detergent, and an oil of lubricating viscosity.

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The additive composition of the present invention may be suitably employed in both manual transmission gear oils and automatic transmission oils. Preferably, the additive composition will be employed in a manual transmission gear oil.

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The present invention further provides a lubricating oil composition comprising a major amount of a transmission oil of lubricating viscosity and an effective synchronizer sticking reducing amount of the additive composition described above. Preferably, the transmission oil is a manual transmission gear oil.

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Among other factors, the present invention is based in part upon the surprising discovery that the unique combination of an oil dispersion of hexagonal boron nitride and a certain viscosity index improver selected from a polymethacrylate, dispersant polymethacrylate and a dispersant olefin copolymer, provides a significant and unexpected reduction in synchronizer sticking when used as an additive composition

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in a manual transmission gear oil.

DETAILED DESCRIPTION OF THE INVENTION

As noted above, the present invention is directed to a novel additive composition for a transmission oil comprising an oil dispersion of hexagonal boron nitride and a

5 viscosity index improver selected the group consisting of:

- a. a polymethacrylate,
- b. a dispersant polymethacrylate, and
- c. a dispersant olefin copolymer;

10 wherein the weight ratio of the oil dispersion of hexagonal boron nitride to the viscosity index improver is in the range of from about 99:1 to about 1:99.

Each of the components in the additive composition of the present invention will be described in further detail below. Unless otherwise stated, all percentages are in

15 weight percent (wt %).

THE OIL DISPERSION OF HEXAGONAL BORON NITRIDE

The additive composition of the present invention contains an oil dispersion of
20 hexagonal boron nitride.

Hexagonal boron nitride, or h-BN, is a hexagonal, graphite-like form of boron nitride, having a layered structure and planar 6-membered rings of alternating boron and nitrogen atoms. On alternate sheets, boron atoms are directly over nitrogen atoms.

25 Hexagonal boron nitride can be prepared by heating boric oxide, boric acid or boric acid salts with ammonium chloride, alkali cyanides or calcium cyanamide at atmospheric pressure. Hexagonal boron nitride may also be prepared by the reaction of boron trichloride or boron trifluoride with ammonia. A discussion of hexagonal boron nitride can be found, for example, in Kirk-Othmer, *Encyclopedia of Chemical*
30 *Technology*, Fourth Edition, Vol. 4, pp. 427-429, John Wiley and Sons, New York, 1992.

Generally, the oil dispersion of hexagonal boron nitride will have a mean particle size of less than 1 micron. Preferably, the oil dispersion of hexagonal boron nitride will have a particle size distribution wherein 90% or greater of the particles are less than about 0.5 microns (500 nanometers, nm), with a preferred mean particle size of less than about 0.3 microns (300 nm).

Typically, the oil dispersion of hexagonal boron nitride will contain from about 1 to about 50 wt % of the hexagonal boron nitride solids, preferably from about 1 to about 20 wt %, and more preferably from about 5 to about 15 wt %, based on the total weight of the oil dispersion.

Preferably, the oil dispersion of hexagonal boron nitride will contain a surfactant as a stabilizer for the oil dispersion. Typical surfactants for use as a stabilizer include ethylene – propylene copolymers, or terpolymers of ethylene, propylene and an unconjugated dienes commonly known as ethylene-propylene-diene terpolymer, ethylene-propylene copolymers grafted with a nitrogen-containing vinyl functionality selected from the group consisting of N-vinyl pyrrolidone and N-vinyl pyridine, and the like. The ethylene-propylene copolymer generally has an average molecular weight in the range of from about 22,000 to about 200,000. A preferred surfactant is ethylene – propylene copolymer which has substantially equal proportions of ethylene and propylene monomers and an average molecular weight of from about 22,000 to about 40,000. When present, the surfactant concentration in the oil dispersion of hexagonal boron nitride will typically range from about 0.1 to about 25 wt %, preferably from about 2 to about 7 wt %, and more preferably from about 3.0 to about 5.0 wt %, based on the total weight of the oil dispersion of hexagonal boron nitride.

The lubricant oil used to prepare the oil dispersion of hexagonal boron nitride may be selected from the same group of natural or synthetic lubricating oils described above for use in preparing the oil dispersion of hydrated alkali metal borate, but other carrier fluids have been found to be satisfactory, including vegetable oils such as rapeseed oil; liquid hydrocarbons such as aliphatic and aromatic naphthas and mixtures thereof; synthetic lubricant fluids such as polyalphaolefins, polyglycols, diester fluids, and mixtures of these liquids. Moreover, the oil used in forming the oil dispersion of

hexagonal boron nitride may be the same as, or different from, the lubricant oil employed in preparing the oil dispersion of hydrated alkali metal borate. Typical oils for preparing the oil dispersion of hexagonal boron nitride include the Group I and Group II base oils, such as 150 solvent neutral petroleum oil.

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In general, the oil dispersion of hexagonal boron nitride is present in the additive composition of the present invention in the range of from about 1 to about 99 wt %, preferably from about 5 to about 95 wt %, and more preferably from about 10 to about 90 wt %, based on the total amount of the additive composition.

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VISCOSITY INDEX IMPROVER (VI IMPROVER)

The additive composition of the present invention contains a polymethacrylate, dispersant polymethacrylate or a dispersant olefin copolymer VI improver.

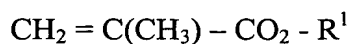
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A. The Polymethacrylate (PMA) or Dispersant Polymethacrylate

Typically, the polymethacrylate VI improvers employed in the present invention are polymeric methacrylates containing short, intermediate, and long-chain hydrocarbon side chains. Short-chain hydrocarbon side chains typically have from about 1 to about 7 carbon atoms. For example, both methyl and butyl (either n-butyl, isobutyl, or mixtures of the two) methacrylates have been used. Methyl methacrylate is the most common. Intermediate-chain hydrocarbon side chains typically contain from about 8 to about 15 carbon atoms and may be derived from alcohols including 2-ethylhexyl alcohol, isodecyl alcohol and alcohol mixtures which may be, for example, C₈ to C₁₀, C₁₂ to C₁₄ or C₁₂ to C₁₅ alcohol mixtures. Long-chain hydrocarbon side chains generally will contain about 14 or more carbon atoms and may be based, for example, on C₁₆ to C₁₈ or C₁₆ to C₂₀ alcohol mixtures.

30 The polymethacrylate VI improvers which may be employed in the present invention are any type of non-dispersant type or dispersant type polymethacrylate compounds which are used as VI improvers for a lubricating oil.

The non-dispersant type polymethacrylate VI improvers may be a polymer of a compound represented by the formula:



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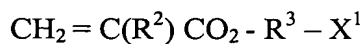
In formula (1) R^1 is a straight chain or branched alkyl group such as methyl, ethyl, propyl, butyl, hexyl, heptyl, octyl, nonyl, decyl, undecyl, dodecyl, tridecyl, tetradecyl, pentadecyl, hexadecyl, heptadecyl, and octadecyl groups.

10 Dispersancy may be incorporated into the PMA with an appropriate polar monomer by any number a methods known by the skilled artisan such as copolymerization, graft polymerization or post reaction of a reactive species into or onto the polymer. Typically, such methods involve the incorporation of a polar group derived from nitrogen or oxygen. Nitrogen-based groups are derived from amines, for example,
15 polyalkyleneamines such as diethylenetriamine and triethylenetetramine. Oxygen-based groups are alcohol-derived such as hydroxyethyl methacrylates or ether-containing methacrylates. Although nitrogen-based PMAs are exemplified in the present invention, oxygen-based PMAs are also contemplated within the scope of the present invention. Examples of oxygen-based PMAs are those derived from
20 polyhydric alcohols such as glycols, trivalent alcohols such as glycerol and higher alcohols, such as erythrytol, pentaerythrytol, mannitol and the like. Moreover, ether-containing PMAs are also well known in the art. Further details of oxygen-based PMAs may be found, for example, in US Patents Nos. 3,249,545 and 3,052,648, the disclosures which is hereby incorporated for all purposes.

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Specific examples of the dispersant polymethacrylate VI improvers are copolymers obtained by copolymerizing one or more monomers selected from compounds represented by formula (1) with one or more nitrogen-containing monomers selected from compounds represented by formulas (2) and (3)

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Formula 2



Formula 3

In formulas (2) and (3) R^2 and R^4 are each independently hydrogen or methyl, R^3 is a
 5 straight chain or branched alkylene group having from about 1 to about 18 carbon
 atoms, such as ethylene, propylene, butylene, pentylene, hexylene, heptylene,
 octylene, nonylene, decylene, undecylene, dodecylene, tridecylene, tetradecylene,
 pentadecylene, hexadecylene, heptadecylene, and octadecylene groups, X^1 and X^2 are
 each independently an amino- or heterocyclic- residue having about 1 or about 2
 10 nitrogen atoms and 0 to about 2 oxygen atoms. Specific examples of X^1 and X^2 are
 dimethylamino, diethylamino, dipropylamino, dibutylamino, anilino, toluidino,
 xylidino, acetylamino, benzoilamino, morpholino, pyrrolyl, pyridyl, methylpyridyl,
 pyrrolidinyl, piperidinyl, quinonyl, pyrrolidonyl, pyrrolidono, imidazolino, and
 pyrazino groups.

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Specific examples of the nitrogen-containing monomers represented by formula (2) or
 (3) are dimethylaminomethylmethacrylate, diethylaminomethylmethacrylate,
 dimethylaminoethylmethacrylate, diethylaminoethylmethacrylate, 2-methyl-5-
 vinylpyridine, morpholinomethylmethacrylate, morpholinoethylmethacrylate, N-
 20 vinylpyrrolidone, and mixtures thereof.

A particularly beneficial PMA employed in the present invention is an ester polymer,
 having principally from about 1 to about 20, preferably from about 8 to about 14,
 carbon atoms. It can be prepared by a polymerization reaction with a basic monomer
 25 and a peroxide or azoic initiator in a hydrocarbon solvent such as toluene or a mineral
 or synthetic base oil. The basic monomers used to prepare the PMA are principally
 monocarboxylic acid esters such as methacrylate, acrylate, crotonate, tiglicate, and
 angelicate. The PMA may also be prepared by reaction with olefinic copolymers (i.e.,
 ethylene-propylene copolymer) in oil.

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The average molecular weight of the PMA will be in the range of from about 20,000
 to about 500,000. Preferably, the molecular weight will range from about 50,000 to
 about 300,000 and more preferably, from about 80,000 to about 150,000.

A further discussion of PMA VI improvers and dispersant PMA VI improvers can be found, for example, in "*Lubricant Additives Chemistry and Applications*", Leslie R. Rudnick, Editor, Chapters 5 and 11, Marcel Dekker, Inc, New York 2003 and U.S. Patent No. 6,642,189.

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B. Dispersant Olefin Copolymer (OCP)

The dispersant OCPs employable in the present invention include copolymers of two or more olefins such as ethylene, propylene, butylene, iso-butylene, isoprene,
10 butadiene and the like, as well as copolymers of these olefins with other monomers such as styrene, cyclopentadiene, dicyclopentadiene, ethylidene-norbornene and so on.

Exemplary dispersant OCPs for the purpose of the present invention relate to ethylene
15 copolymers. Oil soluble ethylene copolymers used in the invention generally will have a number-average molecular weight (M_n) of from above about 5,000 to about 500,000; preferably from about 10,000 to about 200,000 and optimally from about 20,000 to about 100,000. They will generally have a narrow range of molecular weight, as determined by the ratio of weight-average molecular weight (M_w) to
20 number average molecular weight (M_n). Polymers having a M_w / M_n of less than 10, preferably less than 7, and more preferably 4 or less are most desirable. As used herein and (M_n) and (M_w) are measured by the well known techniques of vapor phase osmometry (VPO), membrane osmometry and gel permeation chromatography. In general, polymers having a narrow range of molecular weight may be obtained by a
25 choice of synthesis conditions such as choice of principal catalyst and cocatalyst combination, addition of hydrogen during the synthesis, etc. Post synthesis treatment such as extrusion at elevated temperature and under high shear through small orifices, mastication under elevated temperatures, thermal degradation, fractional precipitation from solution, etc. may also be used to obtain narrow ranges of desired molecular
30 weights and to break down higher molecular weight polymer to different molecular weight grades for VI use.

These polymers are prepared from ethylene and ethylenically unsaturated hydrocarbons including cyclic, alicyclic and acyclic, containing from about 3 to about 28 carbons, e.g. about 2 to about 18 carbons. These ethylene copolymers may contain from about 15 to about 90 wt. % ethylene, preferably from about 30 to about 80 wt. % of ethylene and from about 10 to about 85 wt. %, preferably from about 20 to about 70 wt. % of one or more C₃ to C₂₈, preferably C₃ to C₁₈, more preferably C₃ to C₈, alpha olefins. While not essential, such copolymers preferably have a degree of crystallinity of less than 25 wt. %, as determined by X-ray and differential scanning calorimetry. Copolymers of ethylene and propylene are most preferred. Other alpha-olefins suitable in place of propylene to form the copolymer, or to be used in combination with ethylene and propylene to form a terpolymer, tetrapolymer, etc., include 1-butene, 1-pentene, 1-hexene, 1-heptene, 1-octene, 1-nonene, 1-decene, etc.; also branched chain alpha-olefins, such as 4-methyl-1-pentene, 4-methyl-1-hexene, 4,4-dimethyl-1-pentene, and 6-methylheptene-1, etc., and mixtures thereof.

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The term copolymer as used herein, unless otherwise indicated, includes terpolymers, tetrapolymers, etc., of ethylene, said C₃ to C₂₈ alpha-olefin and/or a non-conjugated diolefin or mixtures of such diolefins which may also be used. The amount of the non-conjugated diolefin will generally range from about 0.5 to about 20 mole percent, preferably from about 1 to about 7 mole percent, based on the total amount of ethylene and alpha-olefin present.

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Representative examples of non-conjugated dienes that may be used as the third monomer in the terpolymer include:

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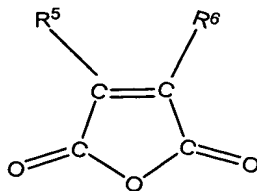
a. Straight chain acyclic dienes such as: 1,4-hexadiene; 1,5-heptadiene; 1,6-octadiene.

b. Branched chain acyclic dienes such as: 5-methyl-1,4-hexadiene; 3,7-dimethyl 1,6-octadiene; 3,7-dimethyl 1,7-octadiene; and the mixed isomers of dihydro-myrcene and dihydro-cymene.

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- c. Single ring alicyclic dienes such as: 1,4-cyclohexadiene; 1,5-cyclooctadiene; 1,5-cyclo-dodecadiene; 4-vinylcyclohexene; 1-allyl, 4-isopropylidene cyclohexane; 3-allyl-cyclopentene; 4-allyl cyclohexene and 1-isopropenyl-4-(4-butenyl) cyclohexane.
- 5 d. Multi-single ring alicyclic dienes such as: 4,4'-dicyclopentenyl and 4,4'-dicyclohexenyl dienes.
- e. Multi-ring alicyclic fused and bridged ring dienes such as: tetrahydroindene; methyl tetrahydroindene; dicyclopentadiene; bicyclo (2.2.1)-hepta 2,5-diene; alkyl, alkenyl, 10 alkylidene, cycloalkenyl and cycloalkylidene norbornenes such as: ethyl norbornene; 5- methylene- 6-methyl-2 -norbornene; 5-methylene-6, 6-dimethyl-2-norbornene; 5-propenyl-2-norbornene; 5-(3-cyclopentenyl)-2-norbornene and 5-cyclohexylidene-2-norbornene; norbornadiene; etc.
- 15 A compound containing at least one ethylenic bond and at least one, preferably two, carboxylic acid groups, or an anhydride group, or a polar group which is convertible into said carboxyl groups by oxidation or hydrolysis may be grafted on the ethylene copolymer. Preferred acid materials are (i) monounsaturated C₄ to C₁₀ dicarboxylic acid wherein (a) the carboxyl groups are vicinyl, i.e., located on adjacent carbon 20 atoms, and (b) at least one, preferably both, of said adjacent carbon atoms are part of said mono unsaturation; or (ii) derivatives of (i) such as anhydrides or C₁ to C₅ alcohol derived mono- or diesters of (i). Upon reaction with the ethylene-alpha-olefin copolymer, the monounsaturations of the dicarboxylic acid, anhydride, or ester becomes saturated. Thus, for example, maleic anhydride becomes a hydrocarbyl 25 substituted succinic anhydride.

Maleic anhydride or a derivative thereof is preferred as it does not appear to homopolymerize appreciably but grafts onto the ethylene copolymer to give two carboxylic acid functionalities. Such preferred materials have the generic formula



wherein R⁵ and R⁶ are the same or different and are hydrogen or a halogen. Suitable examples additionally include chloro-maleic anhydride, itaconic anhydride, or the
 5 corresponding dicarboxylic acids, such as maleic acid or fumaric acid or their monoesters, etc.

As taught by U.S. Patent No. 4,160,739 and U.S. Patent No. 4,161,452, both of which are incorporated herein by reference, various unsaturated comonomers may be grafted
 10 on the ethylene copolymer together with the unsaturated acid component, e.g. maleic anhydride. Such graft monomer systems may comprise one or a mixture of comonomers different from the unsaturated acid component and which contain only one copolymerizable double bond and are copolymerizable with said unsaturated acid component. Typically, such comonomers do not contain free carboxylic acid groups
 15 and are esters containing alpha, beta-ethylenic unsaturation in the acid or alcohol portion; hydrocarbons, both aliphatic and aromatic, containing alpha, beta-ethylenic unsaturation, such as the C₄ to C₁₂ alpha olefins, for example isobutylene, hexene, nonene, dodecene, etc.; styrenes, for example styrene, alpha-methyl styrene, p-methyl styrene, p-sec. butyl styrene, etc.; and vinyl monomers, for example vinyl acetate,
 20 vinyl chloride, vinyl ketones such as methyl and ethyl vinyl ketone, etc. Comonomers containing functional groups which may cause crosslinking, gelation or other interfering reactions should be avoided, although minor amounts of such comonomers (up to about 10% by weight of the comonomer system) often can be tolerated.

25 Specific useful copolymerizable comonomers include the following:

(A) Esters of saturated acids and unsaturated alcohols wherein the saturated acids may be monobasic or polybasic acids containing up to about 40 carbon atoms such as the following: acetic, propionic, butyric, valeric, caproic, stearic, oxalic, malonic,
 30 succinic, glutaric, adipic, pimelic, suberic, azelaic, sebacic, phthalic, isophthalic,

terephthalic, hemimellitic, trimellitic, trimesic and the like, including mixtures. The unsaturated alcohols may be monohydroxy or polyhydroxy alcohols and may contain up to about 40 carbon atoms, such as the following: allyl, methallyl, crotyl, 1-chloroallyl, 2-chloroallyl, cinnamyl, vinyl, methyl vinyl, 1-phenallyl, butenyl, propargyl, 1-cyclohexene-3-ol, oleyl, and the like, including mixtures.

(B) Esters of unsaturated monocarboxylic acids containing up to about 12 carbon atoms such as acrylic, methacrylic and crotonic acid, and an esterifying agent containing up to about 50 carbon atoms, selected from saturated alcohols and alcohol epoxides. The saturated alcohols may preferably contain up to about 40 carbon atoms and include monohydroxy compounds such as: methanol, ethanol, propanol, butanol, 2-ethylhexanol, octanol, dodecanol, cyclohexanol, cyclopentanol, neopentyl alcohol, and benzyl alcohol; and alcohol ethers such as the monomethyl or monobutyl ethers of ethylene or propylene glycol, and the like, including mixtures. The alcohol epoxides include fatty alcohol epoxides, glycidol, and various derivatives of alkylene oxides, epichlorohydrin, and the like, including mixtures.

The components of the graft copolymerizable system are used in a ratio of unsaturated acid monomer component to comonomer component of about 1:4 to about 4:1, preferably about 1.2 to about 2:1 by weight.

Further dispersant functionality may be incorporated into the OCP by reacting with polyamine or polyol, high functionality long chain hydrocarbyl dicarboxylic acid materials having a functionality of from about 1.2 to about 2 and short chain hydrocarbyl substituted dicarboxylic acids, as described, for example, in US Patent No. 5,035,821, which is hereby incorporated for all purposes.

Among the copolymers preferred are ethylene-propylene copolymers (the ratio of ethylene:propylene is preferably about 3:1 to about 1:3), and styrene-isoprene copolymers. Olefin copolymers are manufactured from ethylene and propylene by means of Ziegler catalysts. The molecular weight of olefinic copolymers may vary

widely, but preferred copolymers are those having a molecular weight of from about 30,000 to about 200,000, more preferably from about 40,000 to about 150,000.

Such preferred copolymers include nitrogen atom-containing polymers, for example, those obtained by copolymerizing or grafting, with an acidic component such as maleic acid or anhydride thereof, onto an olefinic copolymer, followed by forming amide or imide linkages by reaction with polyamines.

Another such preferred copolymer is that obtained by oxidizing an olefinic copolymer, followed by reacting the oxidized polymer with polyamines. Still another copolymer is that obtained by oxidizing an olefinic copolymer followed by Mannich condensation with formaldehyde and polyamines.

Another preferred copolymer is that obtained by copolymerizing olefins with a nitrogen atom-containing monomer, or grafting a nitrogen atom-containing monomer onto an olefinic copolymer such as N-vinylpyrrolidone, N-vinylthiopyrrolidone, a dialkylaminoethyl methacrylate or the like (the content of nitrogen atom-containing monomer preferably being from about 0.1 to about 10 wt %).

In general, the VI Improver is present in the additive composition of the present invention in the range of from about 1 to about 99 wt %, preferably from about 5 to about 95 wt %, and more preferably from about 10 to about 90 wt %, based on the total weight of the additive composition.

A further discussion of dispersant OCP VI improvers can be found, for example, in "*Lubricant Additives; Chemistry and Applications*", Leslie R. Rudnick, Editor, Chapters 5 and 10, Marcel Dekker, Inc, New York 2003.

THE HYDRATED ALKALI METAL BORATE

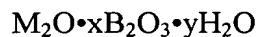
The additive composition of the present invention may optionally further contain an oil dispersion of hydrated alkali metal borate as described below.

Hydrated alkali metal borates are well known in the art. Representative patents disclosing suitable borates and methods of manufacture include: U.S. Patent Nos.

3,313,727; 3,819,521; 3,853,772; 3,912,643; 3,997,454; and 4,089,790.¹⁻⁶

The hydrated alkali metal borates suitable for use in the present invention can be represented by the following general formula:

5



wherein M is an alkali metal, preferably sodium or potassium; x is a number from about 2.5 to about 4.5 (both whole and fractional); and y is a number from about 1.0
10 to about 4.8. More preferred are the hydrated potassium borates, particularly the hydrated potassium triborates. The hydrated borate particles will generally have a mean particle size of less than 1 micron.

In the alkali metal borates employed in this invention, the ratio of boron to alkali
15 metal will preferably range from about 2.5:1 to about 4.5:1.

Oil dispersions of hydrated alkali metal borates are generally prepared by forming, in deionized water, a solution of alkali metal hydroxide and boric acid, optionally in the presence of a small amount of the corresponding alkali metal carbonate. The solution
20 is then added to a lubricant composition comprising an oil of lubricating viscosity, a dispersant and any optional additives to be included therein (e.g., a detergent, or other optional additives) to form an emulsion that is then dehydrated.

Because of their retention of hydroxyl groups on the borate complex, these complexes
25 are referred to as "hydrated alkali metal borates" and compositions containing oil/water emulsions of these hydrated alkali metal borates are referred to as "oil dispersions of hydrated alkali metal borates".

Preferred oil dispersions of alkali metal borates will have a boron to alkali metal ratio
30 of from about 2.5:1 to about 4.5:1. In another preferred embodiment, the hydrated alkali metal borate particles generally will have a mean particle size of less than 1 micron. In this regard, it has been found that the hydrated alkali metal borates

employed in this invention preferably will have a particle size where 90% or greater of the particles are less than 0.6 microns.

5 In the oil dispersion of hydrated alkali metal borate, the hydrated alkali metal borate will generally comprise from about 10 to about 75 wt %, preferably from about 25 to about 50 wt %, more preferably from about 30 to about 40 wt % of the total weight of the oil dispersion of hydrated borate.

10 In general, when employed, the oil dispersion of hydrated alkali metal borate is present in the additive composition of the invention in the range of from about 10 to about 90 wt %, based on the total weight of the additive composition.

The additive compositions and lubricant compositions of the present invention can further employ surfactants, detergents, other dispersants and other conditions as
15 described below and known to those skilled in the art. Optionally, the additive compositions may contain an alkylaromatic or polyisobutenyl sulfonate.

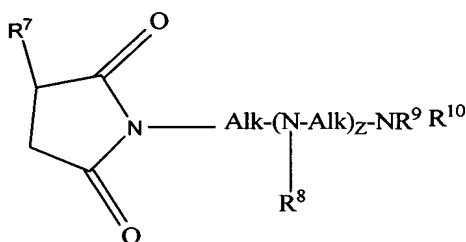
The oil dispersions of hydrated alkali metal borates employed in this invention generally comprise a dispersant, an oil of lubricating viscosity, and optionally a
20 detergent, that are further detailed below.

The dispersant employed in the oil dispersion of hydrated alkali metal borate optionally employable in the present invention can be ashless dispersants such as an alkenyl succinimide, an alkenyl succinic anhydride, an alkenyl succinate ester, and the
25 like, or mixtures of such dispersants.

Ashless dispersants are broadly divided into several groups. One such group is directed to copolymers which contain a carboxylate ester with one or more additional polar function, including amine, amide, imine, imide, hydroxyl carboxyl, and the like.
30 These products can be prepared by copolymerization of long chain alkyl acrylates or methacrylates with monomers of the above function. Such groups include alkyl methacrylate-vinyl pyrrolidinone copolymers, alkyl methacrylate-dialkylaminoethyl methacrylate copolymers and the like. Additionally, high molecular weight amides

and polyamides or esters and polyesters such as tetraethylene pentamine, polyvinyl polyesterates and other polystearamides may be employed. Preferred dispersants are N-substituted long chain alkenyl succinimides.

- 5 Alkenyl succinimides are usually derived from the reaction of alkenyl succinic acid or anhydride and alkylene polyamines. These compounds are generally considered to have the formula



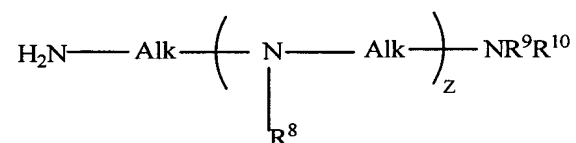
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- wherein R^7 is a substantially hydrocarbon radical having a molecular weight from about 400 to about 3,000, that is, R^7 is a hydrocarbyl radical, preferably an alkenyl radical, containing from about 30 to about 200 carbon atoms; Alk is an alkylene radical of from about 2 to about 10, preferably from about 2 to about 6, carbon atoms, R^8 , R^9 , and R^{10} are selected from a C_1 to C_4 alkyl or alkoxy or hydrogen, preferably hydrogen, and z is an integer from about 0 to about 10, preferably from about 0 to about 3. The actual reaction product of alkylene succinic acid or anhydride and alkylene polyamine will comprise the mixture of compounds including succinamic acids and succinimides. However, it is customary to designate this reaction product as a succinimide of the described formula, since this will be a principal component of the mixture. See, for example, U.S. Patent Nos. 3,202,678; 3,024,237; and 3,172,892.
- 15
- 20

- These N-substituted alkenyl succinimides can be prepared by reacting maleic anhydride with an olefinic hydrocarbon followed by reacting the resulting alkenyl succinic anhydride with the alkylene polyamine. The R^1 radical of the above formula, that is, the alkenyl radical, is preferably derived from a polymer prepared from an olefin monomer containing from about 2 to about 5 carbon atoms. Thus, the alkenyl radical is obtained by polymerizing an olefin containing from about 2 to about 5 carbon atoms to form a hydrocarbon having a molecular weight ranging from about
- 25

400 to about 3,000. Such olefin monomers are exemplified by ethylene, propylene, 1-butene, 2-butene, isobutene, and mixtures thereof.

The preferred polyalkylene amines used to prepare the succinimides are of the
5 formula:



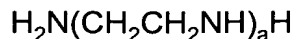
wherein z is an integer of from about 0 to about 10 and Alk, R^8 , R^9 , and R^{10} are as defined above.

10

The alkylene amines include principally methylene amines, ethylene amines, butylene amines, propylene amines, pentylene amines, hexylene amines, heptylene amines, octylene amines, other polymethylene amines and also the cyclic and the higher homologs of such amines as piperazine and amino alkyl-substituted piperazines. They
15 are exemplified specifically by ethylene diamine, triethylene tetraamine, propylene diamine, decamethyl diamine, octamethylene diamine, diheptamethylene triamine, tripropylene tetraamine, tetraethylene pentamine, trimethylene diamine, pentaethylene hexamine, ditrimethylene triamine, 2-heptyl-3-(2-aminopropyl)-imidazoline, 4-methyl imidazoline, N,N-dimethyl-1,3-propane diamine, 1,3-bis(2-
20 aminoethyl)imidazoline, 1-(2-aminopropyl)-piperazine, 1,4-bis(2-aminoethyl)piperazine and 2-methyl-1-(2-aminobutyl)piperazine. Higher homologs such as are obtained by condensing two or more of the above-illustrated alkylene amines likewise are useful.

25 The ethylene amines are especially useful. They are described in some detail under the heading "*Ethylene Amines*" in *Encyclopedia of Chemical Technology*, Kirk-Othmer, Vol. 5, pp. 898-905 (Interscience Publishers, New York, 1950).

The term "ethylene amine" is used in a generic sense to denote a class of polyamines
30 conforming for the most part to the structure



wherein a is an integer from about 1 to about 10.

- 5 Thus, it includes, for example, ethylene diamine, diethylene triamine, triethylene tetraamine, tetraethylene pentamine, pentaethylene hexamine, and the like.

Also included within the term “alkenyl succinimides” are post-treated succinimides such as post-treatment processes involving ethylene carbonate disclosed by
10 Wollenberg, et al., U.S. Patent No. 4,612,132; Wollenberg, et al., U.S. Patent No. 4,746,446; and the like as well as other post-treatment processes each of which are incorporated herein by reference in its entirety.

Preferably, the dispersant component, such as a polyalkylene succinimide, comprises
15 from about 2 to about 40 wt %, more preferably from about 5 to about 20 wt %, and even more preferably from about 5 to about 15 wt %, of the weight of the oil dispersion of hydrated alkali metal borate.

Polyalkylene succinic anhydrides or a non-nitrogen containing derivative of the
20 polyalkylene succinic anhydride (such as succinic acids, Group I and/or Group II mono- or di-metal salts of succinic acids, succinate esters formed by the reaction of a polyalkylene succinic anhydride, acid chloride or other derivative with an alcohol, and the like) are also suitable dispersants for use in the compositions of this invention.

25 The polyalkylene succinic anhydride is preferably a polyisobutenyl succinic anhydride. In one preferred embodiment, the polyalkylene succinic anhydride is a polyisobutenyl succinic anhydride having a number average molecular weight of at least 500, more preferably at least about 900 to about 3,000 and still more preferably from at least about 900 to about 2,300.

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In another preferred embodiment, a mixture of polyalkylene succinic anhydrides is employed. In this embodiment, the mixture preferably comprises a low molecular weight polyalkylene succinic anhydride component and a high molecular weight

- polyalkylene succinic anhydride component. More preferably, the low molecular weight component has a number average molecular weight of from about 500 to below 1,000 and the high molecular weight component has a number average molecular weight of from about 1000 to about 3,000. Still more preferably, both the low and high molecular weight components are polyisobutenyl succinic anhydrides. Alternatively, various molecular weights polyalkylene succinic anhydride components can be combined as a dispersant as well as a mixture of the other above referenced dispersants as identified above.
- As noted above, the polyalkylene succinic anhydride is the reaction product of a polyalkylene (preferably polyisobutene) with maleic anhydride. One can use conventional polyisobutene, or high methylvinylidene polyisobutene in the preparation of such polyalkylene succinic anhydrides. One can use thermal, chlorination, free radical, acid catalyzed, or any other process in this preparation.
- Examples of suitable polyalkylene succinic anhydrides are thermal PIBSA (polyisobutenyl succinic anhydride) described in U.S. Patent No. 3,361,673; chlorination PIBSA described in U.S. Patent No. 3,172,892; a mixture of thermal and chlorination PIBSA described in U.S. Patent No. 3,912,764; high succinic ratio PIBSA described in U.S. Patent No. 4,234,435; PolyPIBSA described in U.S. Patent Nos. 5,112,507 and 5,175,225; high succinic ratio PolyPIBSA described in U.S. Patent Nos. 5,565,528 and 5,616,668; free radical PIBSA described in U.S. Patent Nos. 5,286,799, 5,319,030, and 5,625,004; PIBSA made from high methylvinylidene polybutene described in U.S. Patent Nos. 4,152,499, 5,137,978, and 5,137,980; high succinic ratio PIBSA made from high methylvinylidene polybutene described in European Patent Application Publication No. EP 355 895; terpolymer PIBSA described in U.S. Patent No. 5,792,729; sulfonic acid PIBSA described in U.S. Patent No. 5,777,025 and European Patent Application Publication No. EP 542 380; and purified PIBSA described in U.S. Patent No. 5,523,417 and European Patent Application Publication No. EP 602 863. The disclosures of each of these documents are incorporated herein by reference in their entirety.

Preferably, the polyalkylene succinic anhydride or other dispersant component comprises from about 2 to about 40 wt %, more preferably from about 5 to about 20

wt %, and even more preferably from about 5 to about 15 wt %, of the weight of the oil dispersion of hydrated alkali metal borate.

Typically, in the oil dispersion of hydrated alkali metal borate, the hydrated alkali metal borate is in a ratio of at least 2:1 relative to the polyalkylene succinic anhydride or other dispersant, while preferably being in the range of 2:1 to about 10:1. In a more preferred embodiment the ratio is at least 5:1. In another preferred embodiment, mixtures as defined above of the polyalkylene succinic anhydrides are employed.

- 10 The oil dispersion of hydrated alkali metal borate which is optionally employed in the additive compositions of the present invention may optionally contain a detergent. There are a number of materials that are suitable as detergents for the purpose of this invention. These materials include phenates (high overbased or low overbased), high overbased phenate stearates, phenolates, salicylates, phosphonates, thiophosphonates and sulfonates and mixtures thereof. Preferably, sulfonates are used, such as high overbased sulfonates, low overbased sulfonates, or phenoxy sulfonates. In addition the sulfonic acids themselves can also be used.

- The sulfonate detergent is preferably an alkali or alkaline earth metal salt of a hydrocarbyl sulfonic acid having from about 15 to about 200 carbons. Preferably the term "sulfonate" encompasses the salts of sulfonic acid derived from petroleum products. Such acids are well known in the art. They can be obtained by treating petroleum products with sulfuric acid or sulfur trioxide. The acids thus obtained are known as petroleum sulfonic acids and the salts as petroleum sulfonates. Most of the petroleum products which become sulfonated contain an oil-solubilizing hydrocarbon group. Also included within the meaning of "sulfonate" are the salts of sulfonic acids of synthetic alkyl aryl compounds. These acids also are prepared by treating an alkyl aryl compound with sulfuric acid or sulfur trioxide. At least one alkyl substituent of the aryl ring is an oil-solubilizing group, as discussed above. The acids thus obtained are known as alkyl aryl sulfonic acids and the salts as alkyl aryl sulfonates. The sulfonates where the alkyl is straight-chain are the well-known linear alkylaryl sulfonates.

The acids obtained by sulfonation are converted to the metal salts by neutralizing with a basic reacting alkali or alkaline earth metal compound to yield the Group I or Group II metal sulfonates. Generally, the acids are neutralized with an alkali metal base.

Alkaline earth metal salts are obtained from the alkali metal salt by metathesis.

- 5 Alternatively, the sulfonic acids can be neutralized directly with an alkaline earth metal base. The sulfonates can then be overbased, although, for purposes of this invention, overbasing is not necessary. Overbased materials and methods of preparing such materials are well known to those skilled in the art. See, for example, LeSuer U.S. Patent No. 3,496,105, issued Feb. 17, 1970, particularly columns 3 and 4.

10

The sulfonates are present in the oil dispersion in the form of alkali and/or alkaline earth metal salts, or mixtures thereof. The alkali metals include lithium, sodium and potassium. The alkaline earth metals include magnesium, calcium and barium, of which the latter two are preferred.

15

Particularly preferred, however, because of their wide availability, are salts of the petroleum sulfonic acids, particularly the petroleum sulfonic acids which are obtained by sulfonating various hydrocarbon fractions such as lubricating oil fractions and extracts rich in aromatics which are obtained by extracting a hydrocarbon oil with a selective solvent, which extracts may, if desired, be alkylated before sulfonation by reacting them with olefins or alkyl chlorides by means of an alkylation catalyst; organic polysulfonic acids such as benzene disulfonic acid which may or may not be alkylated; and the like.

20

- 25 The preferred salts for use in the present invention are those of alkylated aromatic sulfonic acids in which the alkyl radical or radicals contain at least about 8 carbon atoms, for example from about 8 to about 22 carbon atoms. Another preferred group of sulfonate starting materials are the aliphatic-substituted cyclic sulfonic acids in which the aliphatic substituents or substituents contain a total of at least 12 carbon atoms, such as the alkyl aryl sulfonic acids, alkyl cycloaliphatic sulfonic acids, the alkyl heterocyclic sulfonic acids and aliphatic sulfonic acids in which the aliphatic radical or radicals contain a total of at least 12 carbon atoms. Specific examples of these oil-soluble sulfonic acids include petroleum sulfonic acids, mono- and poly-

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wax-substituted naphthalene sulfonic acids, substituted sulfonic acids, such as cetyl benzene sulfonic acids, cetyl phenyl sulfonic acids, and the like, aliphatic sulfonic acid, such as paraffin wax sulfonic acids, hydroxy-substituted paraffin wax sulfonic acids, etc., cycloaliphatic sulfonic acids, petroleum naphthalene sulfonic acids, cetyl cyclopentyl sulfonic acid, mono- and poly-wax-substituted cyclohexyl sulfonic acids, and the like. The term "petroleum sulfonic acids" is intended to cover all sulfonic acids that are derived directly from petroleum products.

Typical Group II metal sulfonates suitable for use in the present invention include the metal sulfonates exemplified as follows: calcium white oil benzene sulfonate, barium white oil benzene sulfonate, magnesium white oil benzene sulfonate, calcium dipolypropene benzene sulfonate, barium dipolypropene benzene sulfonate, magnesium dipolypropene benzene sulfonate, calcium mahogany petroleum sulfonate, barium mahogany petroleum sulfonate, magnesium mahogany petroleum sulfonate, calcium triacontyl sulfonate, magnesium triacontyl sulfonate, calcium lauryl sulfonate, barium lauryl sulfonate, magnesium lauryl sulfonate, etc. The concentration of metal sulfonate that may be employed may vary over a wide range, depending upon the concentration of alkali metal borate particles. When present, however, the detergent concentration will generally range from about 0.2 to about 10 wt % and preferably from about 3 to about 7 wt %, based on the total weight of the oil dispersion of hydrated borate. In addition, the compositions of this invention may contain a mixture of both a metal sulfonate and an ashless dispersant, as described above, where the ratio is a factor of achieving the proper stability of the oil dispersion of hydrated alkali metal borate.

The oil of lubricating viscosity used to form the oil dispersions of hydrated alkali metal borate may be any hydrocarbon-based lubricating oil or a synthetic base oil stock. Likewise, these lubricating oils can be added to the oil dispersions and additive compositions containing them, as described herein, in additional amounts, to form finished lubricating oil compositions. The hydrocarbon-based lubricating oils may be derived from synthetic or natural sources and may be paraffinic, naphthenic or aromatic base, or mixtures thereof. The diluent oil can be natural or synthetic, and can be different viscosity grades.

In the oil dispersion of hydrated alkali metal borate, the lubricating oil typically comprises from about 30 to about 70 wt %, more preferably from about 45 to about 55 wt %, based on the total weight of the oil dispersion of hydrated alkali metal borate.

5

When employed the oil dispersion of hydrated alkali metal borate is present in the additive composition of the present invention in the range of from about 1 to about 99 wt %, preferably from about 5 to about 95 wt %, based on the total weight of the additive composition.

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FORMULATIONS

The additive composition of the present invention containing the oil dispersion of hexagonal boron nitride and VI improver, and optionally, the oil dispersion of hydrated alkali metal borate, may be blended further with additional additives to form additive packages containing the present additive compositions. These additive packages typically comprise from about 10 to about 80 wt % of the additive composition of the present invention described above and from about 90 to about 20 wt % of one or more of conventional additives selected from the group consisting of ashless dispersants (0-10 wt %), detergents (0-5 wt %), sulfurized hydrocarbons (0-40 wt %), dialkyl hydrogen phosphates (0-15 wt %), zinc dithiophosphates (0-20 wt %), alkyl ammonium phosphates and/or thio- dithiophosphates (0-20 wt %), phosphites (0 to 10 wt %) fatty acid esters of polyalcohols (0-10wt %), 2,5-dimercaptothiadiazole (0-5 wt %), benzotriazole (0-5 wt %), dispersed molybdenum disulfide (0-5 wt %), foam inhibitors (0-2 wt %), and imidazolines (0-10 wt %) and the like wherein each wt % is based on the total weight of the additive composition.

Fully formulated finished lubricating oil compositions of this invention can be formulated from these additive packages upon further blending with an oil of lubricating viscosity. Preferably, the additive package described above is added to a base oil of lubricating viscosity in an amount of from about 1 to about 40 wt %, preferably from about 2 to about 20 wt %, to provide for the finished lubricating oil

composition wherein the wt % of the additive package is based on the total weight of the lubricating oil composition.

A variety of other additives can be present in lubricating oils of the present invention.

- 5 These additives include antioxidants, rust inhibitors, corrosion inhibitors, extreme pressure agents, antifoam agents, other anti-wear agents, and a variety of other well-known additives in the art.

EXAMPLES

- 10 The invention will be further illustrated by the following examples, which set forth particularly advantageous embodiments. While the examples are provided to illustrate the present invention, they are not intended to limit it.

15 Example 1

- The additive composition of the present invention was evaluated in a lubricating oil for its anti-sticking properties following a test using an SAE No. 2 bench, which evaluates transmission fluids during synchronization. The friction pairs used in this
20 bench comprised a brass synchronizer ring and a steel gear cone.

- During each cycle of the test, the cone is rotating, at a given speed, then the ring moves along the axis of the cone for its braking until it is blocked. At the end of each cycle, the ring is disengaged.

- 25 If sticking occurs, a sticking torque is measured when rotation of the cone is resumed. During the test, the lubricating oil and the metal parts are heated to a temperature between about 60°C and about 90°C. The contact pressure is about 20 MPa and the initial sliding speed is 1.6 m/s.

30

The anti-sticking coefficient for this test was calculated as follows:

$$\text{Anti-sticking coefficient} = 1 - \frac{(\text{No. of cycles with sticking})}{(\text{Total No. of cycles in test})}$$

5

Accordingly, an anti-sticking coefficient of 0 indicates the presence of cone on ring sticking during every cycle of the test. Conversely, an anti-sticking coefficient of 1 indicates no sticking at all was observed over the entire duration of the test. Thus, the higher the anti-sticking coefficient, up to a maximum of 1, the better the anti-sticking performance of the lubricating oil.

10

The test lubricating oil compositions were formulated as follows, all the oils formulated have the same viscosity (about of 9 cSt):

15 Lubricant Composition 1

Lubricant composition 1 was prepared containing the following:

- a) 10 wt % of an oil dispersion of hexagonal boron nitride, wherein the oil dispersion contained about 10 wt % of the hexagonal boron nitride solids, dispersed in a 150 N neutral oil containing a stabilizing agent,
- b) 12 wt % of a long chain polymethacrylate VI Improver sold under the name Viscoplex[®] 0-113 (available from RohMax Additives GmbH, Darmstadt, Germany), and
- c) 78 wt % of a 50/50 mixture of neutral oil (150N plus 600N) and synthetic polyalphaolefin oil.

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Lubricant Composition 2

Lubricant composition 2 was prepared containing the following:

- a) 10 wt % of an oil dispersion of hexagonal boron nitride, wherein the oil dispersion contained about 10 wt % of the hexagonal boron nitride solids, dispersed in a 150 N neutral oil containing a stabilizing agent,

30

- 5 b) 12 wt % of a dispersant long chain polymethacrylate VI Improver sold under the name Viscoplex[®] 0-110 (available from RohMax Additives GmbH, Darmstadt, Germany), and
- c) 78 wt % of a 50/50 mixture of neutral oil (150N plus 600N) and synthetic polyalphaolefin oil.

Lubricant Composition 3

Lubricant composition 3 was prepared containing the following:

- 10 a) 10 wt % of an oil dispersion of hexagonal boron nitride, wherein the oil dispersion contained about 10 wt % of the hexagonal boron nitride solids, dispersed in a 150 N neutral oil containing a stabilizing agent,
- b) 12 wt % of a short chain polymethacrylate VI Improver sold under the name Viscoplex[®] 0-030 (available from RohMax Additives GmbH, Darmstadt, Germany), and
- 15 c) 78 wt % of a 50/50 mixture of neutral oil (150N plus 600N) and synthetic polyalphaolefin oil.

Lubricant Composition 4

20 Lubricant composition 4 was prepared containing the following:

- a) 10 wt % of an oil dispersion of hexagonal boron nitride, wherein the oil dispersion contained about 10 wt % of the hexagonal boron nitride solids, dispersed in a 150 N neutral oil containing a stabilizing agent,
- 25 b) 12 wt % of a dispersant ethylene-propylene olefin copolymer VI Improver with a weight average molecular weight of about 39,000 (Paratone[®] 8500 available from Chevron Oronite Company, LLC, San Ramon, California), and
- c) 78 wt % of a 50/50 mixture of neutral oil (150N plus 600N) and synthetic polyalphaolefin oil.

Lubricant Composition 5

Lubricant composition 5 was prepared containing the following:

- a) 7 wt % of an oil dispersion of hydrated potassium triborate, wherein the oil dispersion contained about 30 wt % of the hydrated potassium triborate, dispersed in a 150 N neutral oil,
- 5 b) 10 wt % of an oil dispersion of hexagonal boron nitride, wherein the oil dispersion contained about 10 wt % of the hexagonal boron nitride solids, dispersed in a 150 N neutral oil containing a stabilizing agent,
- c) 12 wt % of a long chain polymethacrylate VI Improver sold under the name Viscoplex[®] 0-113 (available from RohMax Additives GmbH, Darmstadt, Germany), and
- 10 d) 71 wt % of a 50/50 mixture of neutral oil (150N plus 600N) and synthetic polyalphaolefin oil.

Lubricant Composition A (comparative)

Comparative lubricant composition A was prepared containing the following:

15

- a) 7 wt % of an oil dispersion of hydrated potassium triborate, wherein the oil dispersion contained about 30 wt % of the hydrated potassium triborate, dispersed in a 150 N neutral oil, and
- b) 93 wt % of a 50/50 mixture of neutral oil (150N plus 600N) and synthetic
- 20 polyalphaolefin oil.

Lubricant Composition B (comparative)

Comparative lubricant composition B was prepared containing the following:

- 25 a) 10 wt % of an oil dispersion of hexagonal boron nitride, wherein the oil dispersion contains about 10 wt % of the hexagonal boron nitride solids, dispersed in a 150 N neutral oil containing a stabilizing agent, and
- b) 90 wt % of a 50/50 mixture of neutral oil (150N plus 600N) and synthetic
- 30 polyalphaolefin oil.

Lubricant Composition C (comparative):

Comparative lubricant composition C was prepared containing the following:

- 5
- a) 10 wt % of an oil dispersion of hexagonal boron nitride, wherein the oil dispersion contained about 10 wt % of the hexagonal boron nitride solids, dispersed in a 150 N neutral oil containing a stabilizing agent,
 - b) 12 wt % of a non-dispersant type ethylene-propylene olefin copolymer VI Improver with a weight average molecular weight of about 90,000 (Paratone[®] 8002 available from Chevron Oronite Company, LLC, San Ramon, California), and
 - c) 78 wt % of a 50/50 mixture of neutral oil (150N plus 600N) and synthetic polyalphaolefin oil.

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Lubricant Composition D (comparative):

Comparative lubricant composition D was prepared containing the following:

- 15
- a) 10 wt % of an oil dispersion of hexagonal boron nitride, wherein the oil dispersion contained about 10 wt % of the hexagonal boron nitride solids, dispersed in a 150 N neutral oil containing a stabilizing agent,
 - b) 0.5 wt % of a polyisobutenyl mono-succinimide, and
 - c) 89.5 wt % of a 50/50 mixture of neutral oil (150N plus 600N) and synthetic polyalphaolefin oil.

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Lubricant Composition E (comparative):

Comparative lubricant composition E was prepared containing the following:

- 25
- a) 10 wt % of an oil dispersion of hexagonal boron nitride, wherein the oil dispersion contained about 10 wt % of the hexagonal boron nitride solids, dispersed in a 150 N neutral oil containing a stabilizing agent
 - b) 0.5 wt % of a polyisobutenyl bis-succinimide, and
 - c) 89.5 wt % of a 50/50 mixture of neutral oil (150N plus 600N) and synthetic polyalphaolefin oil.

Table 1

Sample	No. of Cycles with Cone on Ring Sticking	Total No. of Cycles	Anti-sticking coefficient
Base oil	5000	5000	0
Comparative Composition A	8100	8100	0
Comparative Composition B	6600	6600	0
Comparative Composition C	5700	5700	0
Comparative Composition D	6400	6400	0
Comparative Composition E	8100	8100	0
Composition 1	500	7100	0.93
	1050	5600	0.81
Composition 2	100	6800	0.99
Composition 3	1200	6850	0.82
Composition 4	200	20000	0.99
Composition 5	650	20000	0.97

5 The above data demonstrates that the additive composition of the present invention provides significant anti-sticking performance and shows a marked improvement over the comparative compositions.

10 From the foregoing description, various modifications and changes in the above-described invention will occur to those skilled in the art. All such modifications coming within the scope of the appended claims are intended to be included therein.